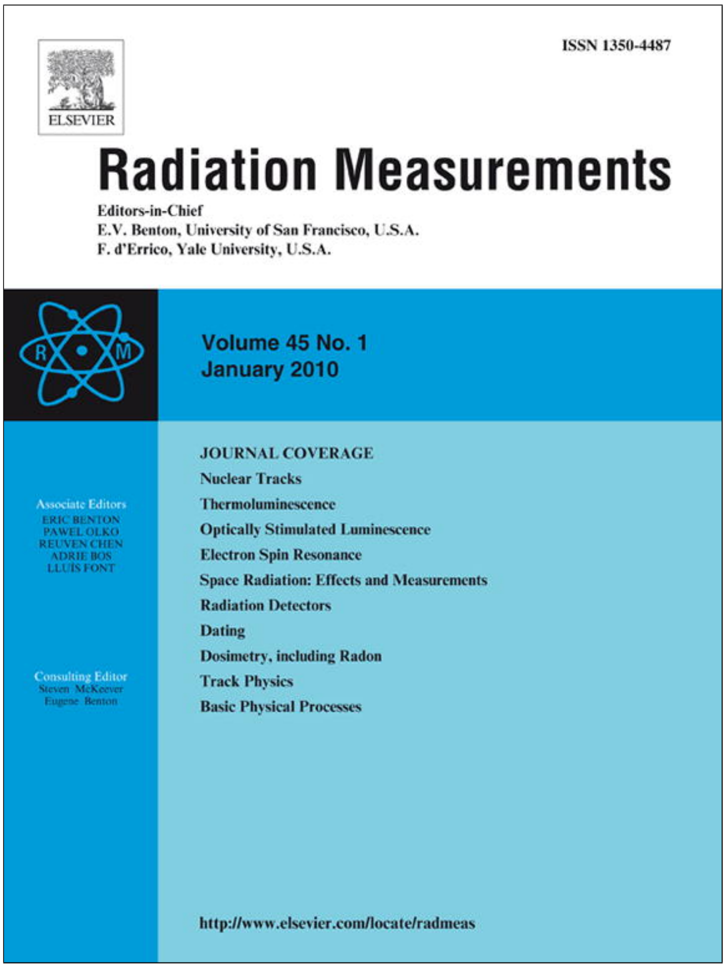
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The effects of low ambient temperatures on optically stimulated luminescence (OSL) processes: Relevance to OSL dating of martian sediments

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A B S T R A C T

Advances in luminescence dosimetry have made geochronological dating of materials from extreme environments possible through the use of optically stimulated luminescence (OSL) single-aliquot techniques. However, these environments present challenges not always encountered in routine OSL dating, such as sediments that have been stored at low, and possibly highly variable, ambient temperatures. In recent years OSL has been proposed as a method for dating recent depositional events on surfaces of other planets, specifically, Mars. As a result it has become necessary to examine the constraints that may be imposed on the OSL method by the extreme environments of extraterrestrial planetary bodies. In this paper we report on investigations of the possible effects a low storage and/or a low OSL measurement temperature could have on the OSL process and the subsequent results. Pertinent OSL properties include the stability of electron traps, the overall luminescence efficiency, and possible thermal assistance processes. The particular focus of the work is on the potential application of the OSL technique for dating surface sediments on Mars. We report the results of OSL experiments on martian simulant materials, and of generalized computer simulations of potential OSL behavior. It is concluded that the stimulation and irradiation/calibration temperatures need to be maintained fixed throughout the experiment – i.e. the dose estimation process – and that the temperature during OSL stimulation needs to be appreciably higher than the highest temperature experienced during natural irradiation. The consequences of these findings for establishing an OSL protocol and instrument package for dating martian regolith material are discussed.

1. Introduction

OSL dating has been successfully applied to materials from a wide range of environments and situations (e.g. Roberts, 1997; Murray and Olley, 2002; Fattahi and Stokes, 2003). As the technology and expertise within the OSL dating field has continued to expand, the technique is being applied to, or is being discussed for, new depositional environments and materials such as fulgurites (Navarro-Gonźalez et al., 2007), exposure of stone surfaces (Greilich and Wagner, 2006), and sediments from ice cores (Lepper et al., 2001). The technique has also been suggested for in situ dating of martian regolith (Lepper and McKeever, 2000; McKeever et al., 2003; Doran et al., 2004). The latter goal faces many challenges (Kalchgruber et al., 2007). Among them are included issues related to estimation of the natural dose rate (De Angelis et al., 2006; Dartnell et al., 2007a,b; Banerjee and Dewangan, 2008), the need to work with unseparated polymineralic samples of uncertain mineral content (Kalchgruber et al., 2006; Blair et al., 2007), fading of the luminescence signal from basaltic materials (Banerjee et al., 2002; Tsukamoto and Duller, 2008), a thin atmosphere with a higher ultraviolet content compared to the Earth (McKeever et al., 2006), an environment of low and variable ambient temperature and, of course, the need to design a robotic instrument to perform the dating procedure in situ, rather than in a laboratory (McKeever et al., 2003, 2006; Jain et al., 2006; Kalchgruber et al., 2007). This paper focuses on one of the challenges to developing robotic in situ dating for the martian surface and sub-surface, namely that related to the ambient temperature.

Although OSL dating techniques are being extended to a wide variety of terrestrial environmental conditions, from cold icy deposits to hot desert climates, most minerals that are dated by OSL are found in more benign conditions with temperatures varying annually over a maximum range of perhaps 50 K for the entirety of their storage period. However, on Mars the average ambient temperature is much lower than that normally found on Earth with much larger diurnal and annual variations. At the Viking 1 landing

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site (22°N latitude) the diurnal temperature variations were found to be from approximately 150–240 K (Hansson, 1997), while Kieffer estimated diurnal variations at the Viking 2 site (44°N) from 185 K to 265 K in the martian summer, and 145–190 K in winter, with overall annual variations, therefore, from 145 K to 265 K (Kieffer, 1976). The global variation is reported to be from 140 K to 300 K (Kieffer et al., 1992). Furthermore, Kieffer (1976) calculates that the diurnal fluctuation does not penetrate beyond about 25 cm into the regolith and that the temperature at depth differs negligibly from the average surface temperature. The latter follows the average annual variation, from approximately 160 K to 225 K.

Lower and widely variable storage temperatures such as those found on Mars may have profound implications for the procedures used to recover the natural radiation dose using OSL and for the design of a suitable robotic OSL instrument. Possible issues include the stability of some traps that, on Earth, would normally be considered unstable and very different pre-heating scenarios that may be called for as a result. Furthermore, if the robotic instrument was unable to have onboard heating (due to power constraints) and instead was required to operate at ambient temperatures, then the efficiency of optical stimulation from the traps could be decreased (due to a reduction in thermally assisted optical transitions), the probability of recombination might be reduced (due to greater competition from shallow traps), while the efficiency of luminescence emission during recombination could, conversely, be increased (due to a reduction in thermal quenching). Operation of the instrument at different ambient temperatures could, therefore, cause significant problems in calibration and analysis such that the operational utility of the instrument could be compromised. Consequently, it becomes important to assemble an understanding of how the martian ambient conditions may impact the operation and design of a robotic dating instrument in order that an instrument capable of performing the necessary OSL protocols can be designed. Each of these issues is discussed in more detail below.

1.1. Pre-heating and trap stability

In OSL dating procedures, a preheat is normally employed to isolate traps that are stable over the geologic period of interest. For terrestrial samples such traps typically give rise to TL peaks above approximately 470 K when the mineral is heated following irradiation. However, at lower ambient temperatures, other traps that are normally considered unstable could now be geologically stable, and traps corresponding to much lower TL peak temperatures may become important in the charge trafficking process. This in turn can mean that the OSL preheat temperature for martian samples may be considerably lower than that typically used in the laboratory on Earth, thus conserving both time and instrument power. In addition, the measurement temperature is often chosen sufficiently high so that those optically sensitive traps that are unstable during natural irradiation (and therefore do not contribute to the natural OSL signal) do not take part in any significant way in the OSL process whereby charge liberated by optical stimulation cannot be retrapped by these unstable traps. Therefore, if minerals that have been stored at lower temperatures on Mars are to be used for OSL dating purposes, pre-heating procedures and temperatures, and OSL measurement temperatures may have to be adjusted and optimized.

1.2. OSL efficiency

The optical stimulation process is often found to be temperature dependent (Hütt and Jaek, 1993; Spooner, 1994). While optical stimulation is the primary way that charge is evicted from traps in OSL dating, the process is often thermally assisted and optical stimulation at an elevated temperature will more effectively evict charge from the traps resulting in a faster OSL decay. Conversely, the process may be less efficient if the optical stimulation is carried out at reduced temperatures. This could have an impact on both equivalent dose estimation and the time necessary to bleach the samples in nature (i.e. the efficiency of 'zeroing' the OSL signal).

In addition to the possibility of reduced stimulation efficiency there is the possibility of a reduction in the probability of recombination during OSL measurement due to the re-trapping of the released charge carriers in traps that normally might be considered too unstable to participate in the process. This was noted above in the discussion of pre-heating but it should also be mentioned here as a possible process by which the recombination efficiency is reduced. By recombination efficiency we mean the number of charges stimulated from traps versus the number of radiative recombination events they subsequently undergo during optical stimulation. If a proportion of the released electrons is "lost" to traps, then these electrons are no longer available to take part in recombination. This effect, and the above effect of reduced stimulation efficiency, can be countered by performing the experiment at an elevated temperature.

Opposing the above negative effects of a low OSL measurement temperature is the potential for an increase in the luminescence efficiency – i.e. an increase in the number of photons emitted as luminescence for a given number of electron-hole recombination events. As the temperature is decreased one might expect a decrease in the probability of non-radiative relaxation of electrons from excited states in the recombination center. The opposite effect to this (i.e. an increase in the non-radiative recombination probability with an increase in temperature) is known as "thermal quenching". This phenomenon is quite widespread among luminescent minerals and is normally described by the Mott-Seitz law, as used by Wintle (1975) in a study of thermal quenching in quartz, viz:

$$\eta = \frac{1}{1 + C\exp\left(\frac{-W}{k_B T}\right)} \tag{1}$$

where η is the luminescence efficiency, C is a constant, W is the thermal activation energy (eV), k_B is Boltzmann's constant (eV K^{-1}), and T is the temperature (K). The thermal activation energy varies by material and is defect-specific. Measurements at lower temperatures could reveal new aspects of thermal quenching for many materials of interest in luminescence dating.

Considering the ways in which the luminescence process can be affected by the ambient temperature as well as by the temperature at which the OSL measurements and calibrations are carried out, research into the properties of minerals irradiated and stimulated at low temperatures is necessary for developing OSL dating protocols for Mars. The following sections will describe a system and some initial experiments that have been designed to address these issues. Results of simulations of OSL by numerical methods will also be presented to gain further insight into the processes involved.

2. Equipment and materials for experiments

The low-temperature TL/OSL system (Fig. 1; Blair et al., 2006) used for this work was designed to control the sample temperature over the range from 120 K to 470 K by using an Omega CN3251 temperature controller connected to a manual liquid nitrogen pump and two 50 W pencil heaters (Watlow Inc.). In order to avoid condensation when cooling the sample, the system is maintained at a vacuum of approximately 5×10^{-4} Torr using a turbomolecular

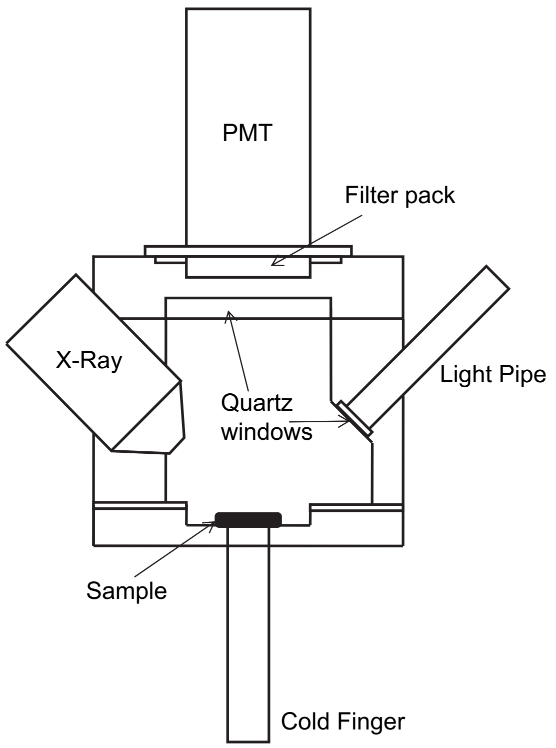

Fig. 1. Diagram of the low-temperature TL/OSL system.

pump. Sample irradiation is accomplished by a 40 kVp Moxtek X-ray tube operating at 35 kV and 100 μA delivering 69 ± 4 mGy/s at the sample. (The reference calibration source was a ^{60}Co secondary standard at the National Institute for Science and Technology, using Al_2O_3:C OSL chips as dosimeters.) Optical stimulation through a quartz window was via a 100 mW Diode Pumped Solid State (DPSS) laser (532 nm) from Extreme Lasers Inc. (USA) operating in continuous-wave mode. The laser delivered approximately 10 mW cm^{-2} at the sample position via a liquid light guide. The radioluminescence (RL), thermoluminescence (TL), or OSL signals were detected through a filter pack containing UV transmitting Hoya U-340 filters (transmission between 290 nm and 390 nm) and a photomultiplier tube operated in photon-counting mode using a Standford Research SR400 photon counter.

The luminescence properties of two martian soil simulants were studied. The simulants, named OSU Mars-1 and OSU Mars-2, were developed at Oklahoma State University (OSU) based on spectroscopic data from the Thermal Emission Spectrometer (TES) onboard the Mars Global Surveyor (MGS) spacecraft (Christensen et al., 2001; Bandfield et al., 2000; Bandfield, 2002). Both simulants consist of plagioclase feldspars, pyroxenes, and hematite, and OSU Mars-2 also contains a significant amount of obsidian. Other research (Kalchgruber et al., 2006) indicates that the majority of the OSL signal (both blue and infrared stimulated) derives from the plagioclase and pyroxene components. The detailed composition of these simulants is given in Table 1, and further details on the composition of the simulants can be found in Kalchgruber et al. (2006).

3. OSL properties of materials irradiated and stimulated at low temperatures

3.1. Reproducibility

Samples were first bleached for 300 s with blue light (to remove any pre-existing charge from traps) and then subjected to 5 cycles of irradiation (5 Gy each cycle) and OSL stimulation (300 s) while maintained at a temperature of 298 K. The resulting OSL curves overlap each other, and the OSL intensity (total area under the curve above the background level) varied by only 4% for OSU Mars-1 and 5% for OSU Mars-2.

Table 1
Mineral abundances of two martian soil simulants, OSU Mars-1 and OSU Mars-2. The table lists mineral groups that may be present on Mars, and the weight percent of each mineral is given.

Group/mineral	OSU Mars-1 (%)	OSU Mars-2 (%)
Plagioclase		
Bytownite	21.67	15
Andesine	21.67	15
Labradorite	21.67	15
Pyroxene		
Augite	15	5
Diopside	15	5
Obsidian	0	40
Hematite	5	5

3.2. Luminescence efficiency

The luminescence efficiency was studied by monitoring RL under constant irradiation as the samples were cooled from room temperature to 170 K. RL is produced during irradiation when electrons in the conduction band recombine with holes at recombination centers and reflects the luminescence efficiency of the material. To ensure that changes in RL intensity were due to temperature changes, the RL signal was allowed to come to a constant level and the RL signal was taken as the average over 10 s at this constant level. The results, normalized to the RL at 273 K, are presented in Fig. 2. Both samples show an increase (20–30%) in RL with decreasing temperature.

The observed higher luminescence efficiency at low temperatures is most easily explained by thermal quenching. Significant thermal quenching has been reported in the literature for feldspar samples (Duller, 1997; White et al., 1986; Vicosekas et al., 1994; Barnett and Bailiff, 1997), but most of those experiments were conducted on orthoclase specimens and used detection windows with wavelengths greater than 400 nm. In the current experiments more than one recombination center (wavelength) may be monitored with the detection window used. The step-like shape of the data in Fig. 2 supports the notion that at least 2 recombination centers are involved. In a general sense, therefore, the decrease in luminescence efficiency with increasing temperature may be due

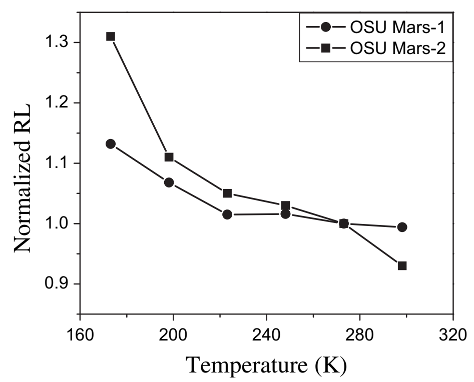

Fig. 2. Normalized RL for OSU Mars-1 and OSU Mars-2 as indicated. The RL was normalized to the value at 273 K.

to multiple recombination centers, some of which may not even contribute to luminescence at room temperature (or above) due to quenching. As a result of the broad emission window used for these experiments no specific conclusions as to the cause of increased luminescence efficiency with decreasing temperatures can be made over and above the general conclusions noted here, but such detailed analysis is not our current purpose.

3.3. Thermoluminescence

3.3.1. Low-temperature TL

The samples were irradiated at 170 K (10 Gy) and heated to room temperature at a heating rate of approximately 0.3 K/s. The solid lines in Fig. 3(a) show the resulting TL from these experiments. Both of the martian soil simulants show TL peaks near 240 K, and OSU Mars-1 shows an additional TL peak near 280 K. Although it is difficult to determine the number of components of these TL curves, these low-temperature peaks indicate the presence of trapping states that are relatively stable at temperatures below approximately 200 K and unstable at higher temperatures.

To examine the optical sensitivity of these trapping centers, the same procedure was repeated, but a bleach for 300 s was performed using the previously described 532 nm DPSS laser after irradiation at 173 K and before heating to record the TL. Comparing the TL curve after bleaching (dashed lines, Fig. 3) with the original TL curve (solid lines), it can be seen that the previously identified trapping levels are optically sensitive. The low-temperature traps for the martian soil simulants were only partially emptied using the current bleaching conditions, however, and these traps may not be a significant source of OSL.

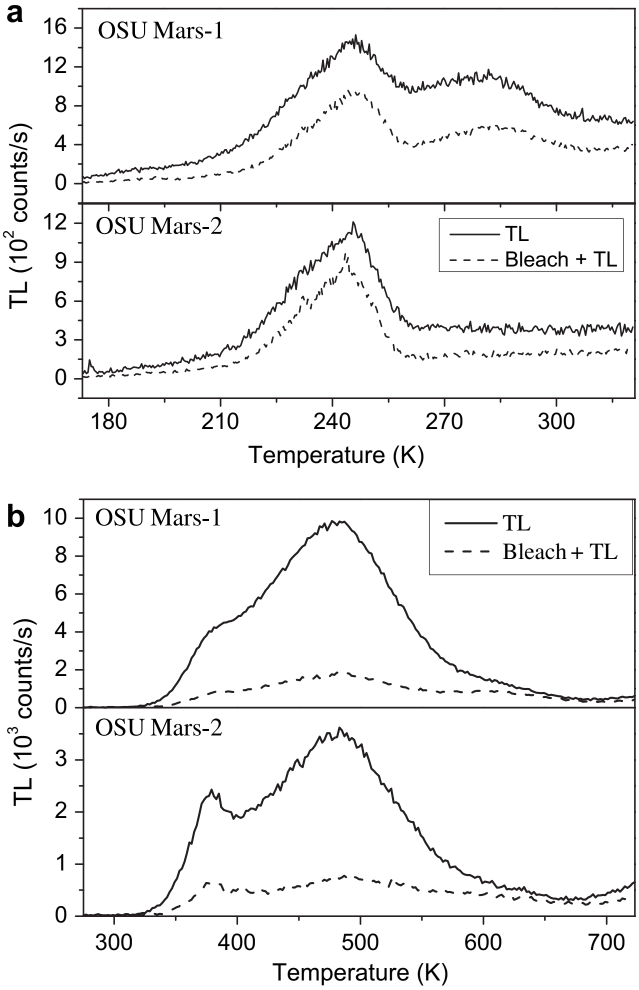

Fig. 3. (a) TL from OSU Mars-1 and OSU Mars-2 after irradiation at 173 K. The solid line represents the TL measured 300 s after irradiation, and the dashed line represents the TL measured after irradiation and bleaching. (b) TL from OSU Mars-1 and OSU Mars-2 after irradiation at 298 K. The solid line represents the TL measured 300 s after irradiation, and the dashed line represents the TL measured after irradiation and 300 s bleaching.

3.3.2. Above-room temperature TL

The low-temperature system described in this paper was not capable of recording glow curves substantially above room temperature. To do so we used a Risø TL/OSL-DA-15 system with 470 nm blue diodes, delivering 31 mW cm^{-2} at sample position and U-340 detection filters. Fig. 3(b) shows the above-RT-TL for OSU Mars-1 and Mars-2. The dose delivered was 40 Gy and the dose rate was 0.1 Gy/s at 298 K. The solid line shows the TL signal with a 300 s delay after irradiation, the dashed line was obtained with a 300 s/470 nm bleach after irradiation. The glow curves consist of several overlapping peaks with two dominant ones at approximately 370 K and 480 K. All trapping levels are optically sensitive in that they bleach freely under the conditions of this experiment.

3.4. Effect of stimulation and irradiation temperatures

The preliminary measurements noted so far were precursors to the main study, which was to examine the general effects of varying irradiation and stimulation temperatures on the OSL properties of the martian soil simulants. These effects were studied by: (a) fixing the irradiation temperature and varying the OSL stimulation temperature; (b) varying the irradiation temperature and fixing the stimulation temperature; and (c) varying both the irradiation and stimulation temperatures together.

The effect of stimulation temperature on the OSL measurements was investigated by irradiating the samples (5 Gy) at room temperature and performing the OSL readout at different temperatures between room temperature and 173 K. Some representative OSL curves are plotted in Fig. 4, the insets showing the normalized integral OSL intensities as a function of stimulation temperature. For both simulants we observe a decrease in OSL intensity with decreasing stimulation temperature, with a step-like feature near 240 K. When considered along with the data of Fig. 2, which suggests an increase in luminescence efficiency as the temperature is lowered, these results may seem surprising. However, as the OSL measurement temperature is lowered, low-temperature trapping states become competitors to the recombination process (as can be inferred from the TL curves of Fig. 3(a)) and this effect appears to dominate over the luminescence efficiency enhancement. The two regions of OSL decrease observed in Fig. 4 correspond to the increasing stability of the traps that yield TL in the 270–300 K region, and the traps yielding TL in the 200–270 K region (Fig. 3(a)).

The effect of irradiation temperature on OSL production was studied by irradiating the samples (5 Gy) at various temperatures from 173 K to 298 K while always measuring OSL at 298 K. The OSL curves and integral OSL signals are shown in Fig. 5. Neither sample showed a substantial change in the integrated OSL signal as the irradiation temperature was lowered (20% decrease for OSU Mars-1; 22% increase for OSU Mars-2). In both cases an initial decrease is observed followed by an increase in integrated OSL as the temperature is reduced. The initial decrease is probably a result of traps near 280–300 K becoming more effective at trapping charge as the irradiation temperature is lowered, with this effect being larger for OSU Mars-1. The latter sample shows a more intense TL peak near these latter temperatures (Fig. 3(a)). An explanation for the subsequent increase, however, is less obvious at this stage and

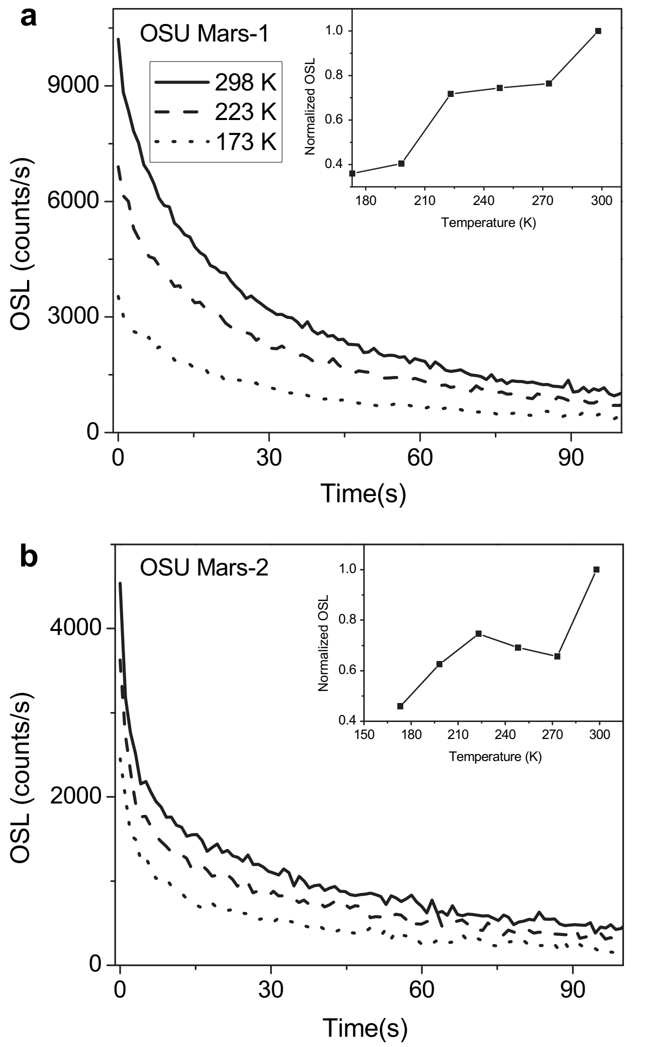

Fig. 4. OSL from samples OSU Mars-1 (a) and OSU Mars-2 (b) irradiated at 298 K and stimulated at various temperatures as described in the text. OSL decay curves for various temperatures are shown, and the insets show the normalized integrated OSL signals versus stimulation temperature. Normalization was at 298 K.

is left until a discussion of the computer simulations in a later section.

We also varied the irradiation and stimulation temperatures together. For this, the samples were cooled to the specified temperature and bleached for 300 s. This was followed by irradiation (5 Gy) and OSL measurement at the same temperature. Fig. 6 shows that the OSL output at first decreases and then increases with decreasing temperature. This observation is also discussed later.

3.5. *Dose recovery*

"Dose recovery" refers to the ability to determine ("recover") a known dose delivered in the laboratory (Murray and Wintle, 2003). Samples of OSU Mars-1 and OSU Mars-2 were given 5 Gy doses, and the single-aliquot regeneration (SAR) procedure (Murray and Wintle, 2000) was used. The calculated dose divided by the actual delivered dose is known as the "dose recovery ratio". Ideally, this ratio should equal 1.0. Regeneration doses of 4, 5, and 6 Gy were used in the SAR procedure, along with a test dose of 1.25 Gy. Intentionally, formal preheats (at a fixed temperature for a fixed time) were not used, for two reasons. Firstly, some of the

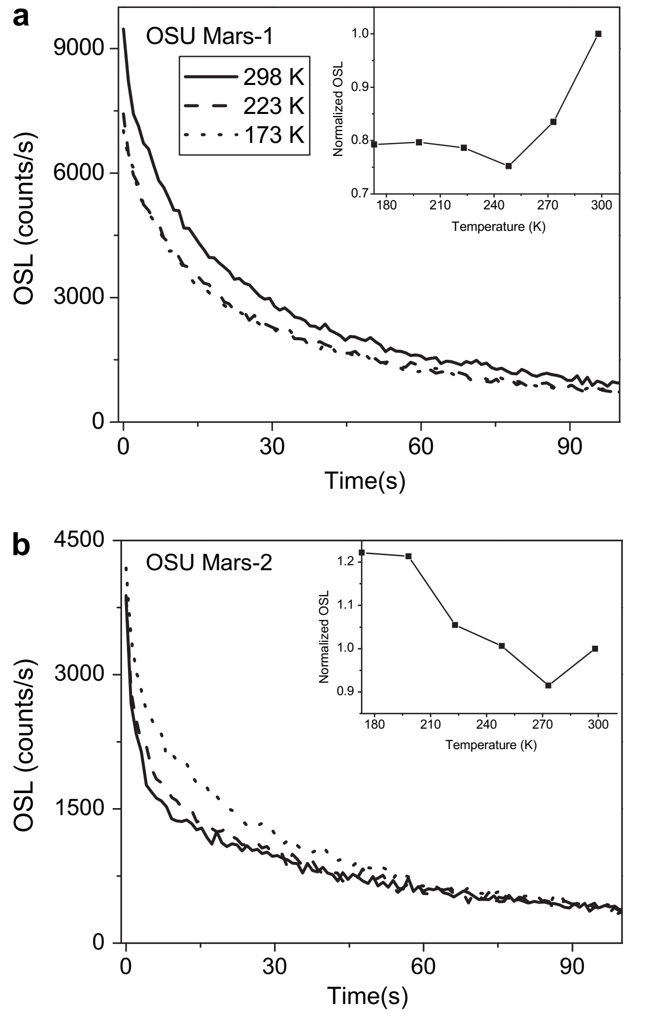

Fig. 5. OSL from samples OSU Mars-1 (a) and OSU Mars-2 (b) that have been irradiated at various temperatures and stimulated at 298 K as described in the text. The insets show the normalized integrated OSL signals versus irradiation temperature.

irradiation/stimulation scenarios to be examined included irradiation at low temperature with stimulation at high temperatures. This is, in effect, a preheat since traps stable during irradiation but unstable during the OSL measurement would be emptied by the increase in temperature between the irradiation and stimulation periods. Secondly, power constraints on the robotic spacecraft will be considerable and this biased us against using elevated temperatures for lengthy periods as much as possible. We specifically wished to determine the accuracy of the SAR procedure in these circumstances. While perhaps not an ideal OSL dating protocol, we nevertheless wished to examine the effects, adverse or otherwise, of this procedure on the final accuracy of the recovered dose, for the reasons stated above.

Several different combinations of irradiation and OSL measurement temperature were used, including an experiment conducted completely at 298 K that was treated as a baseline or "control" experiment. The details of the procedural parameters, along with the obtained "dose recovery ratios" are given in Table 2. In two experiments (5 and 6), the known dose was delivered in stages at three different temperatures in a crude simulation of the temperature variation on Mars.

Although not every conceivable combination of irradiation and optical stimulation temperatures can be tested in such experiments, it may be inferred from the data of Table 2 that the

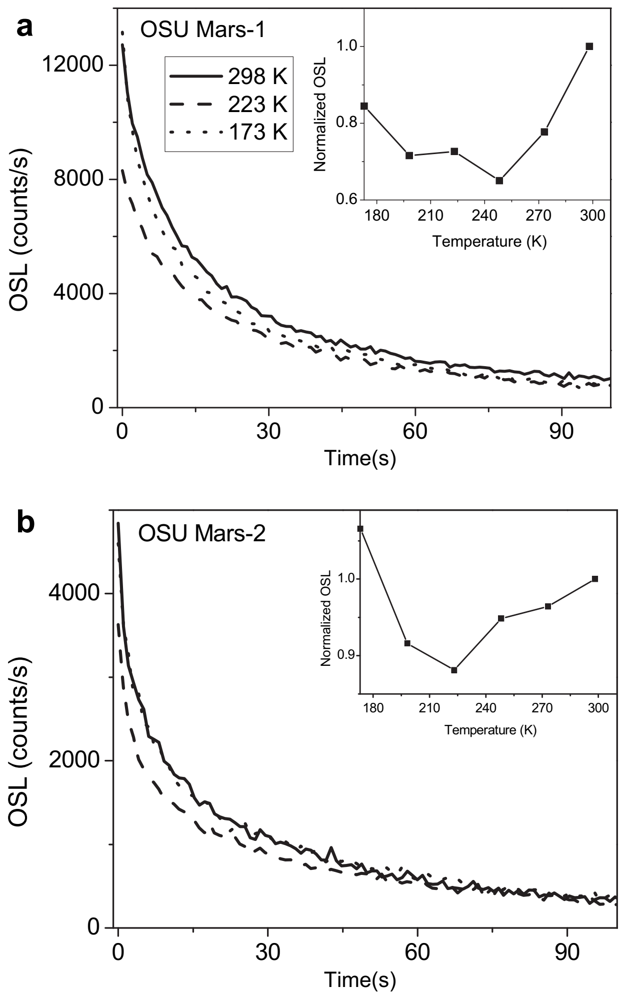

Fig. 6. OSL from samples OSU Mars-1 (a) and OSU Mars-2 (b) that have been irradiated and stimulated at the same temperature as described in the text. The insets show the normalized integrated OSL signals versus temperature.

stimulation temperature (T_{OSL}) must be equal to or greater than the maximum temperature that the sample experienced during irradiation (T_{irr}). In the two cases where the OSL stimulation temperature was lower than the maximum temperature during the irradiation (experiment numbers 4 and 5), there was a significant underestimation of the known dose presumably due to charge being retrapped in low-temperature traps during optical stimulation. Note that pre-heating would be irrelevant in this case since the sample had already experienced a high temperature during irradiation before being cooled to low temperature for stimulation. Similarly, pre-heating for cases 3 and 6 would also be of little relevance since the stimulation temperature is much higher than the irradiation temperature.

4. Numerical simulations of natural materials

Although characterization studies of materials and dose recovery tests involving irradiation and stimulation at low temperatures are necessary and instructive, there is a major limitation of these studies. We currently cannot conduct luminescence experiments on materials that have been naturally irradiated at low temperatures without first heating the samples. Even sediments from ice cores (Lepper et al., 2001) must first be brought into the lab and heated to at least room temperature before luminescence

Table 2
Dose recovery experiments conducted in the low-temperature OSL system. The temperatures of the known dose irradiation ($T_{irr,k}$), the regeneration (and test) dose irradiations ($T_{irr,r}$), and the OSL measurement (T_{OSL}) are given along with the dose recovered ratio for OSU Mars-1 and OSU Mars-2. (Dose ratio = recovered dose/administered dose). Note that in experiments 5 and 6 the known dose was delivered in stages at three different temperatures to simulate the diurnal temperature variation on Mars.

Exp. #	$T_{irr,k}$ (K)	$T_{irr,r}$ (K)	T_{OSL} (K)	Dose ratio OSU Mars-1	Dose ratio OSU Mars-2
1	298	298	298	1.01 ± 0.25	1.02 ± 0.14
2	173	173	173	1.07 ± 0.77	0.98 ± 0.11
3	173	298	298	1.01 ± 0.04	0.94 ± 0.32
4	298	173	173	0.33 ± 0.64	0.26 ± 0.01
5[a]	223 298 173	173	173	0.60 ± 0.09	0.39 ± 0.15
6[a]	223 298 173	298	298	0.95 ± 0.52	1.04 ± 0.28

[a] 1.7 Gy delivered at each temperature.

measurements can be made. On Mars, performing experiments at the ambient temperature would be advantageous from a power consumption and engineering point of view (McKeever et al., 2003). Therefore, the influences of low storage temperature during natural irradiation and variable temperature during the dose estimation process need to be studied.

One way to examine possible protocols to be used in a dating procedure is to numerically simulate natural irradiation of a sample and the subsequent methodology used in an OSL dating routine. Not only do numerical simulations aid in determining the best experimental steps for a dose recovery procedure, they also augment the understanding of the luminescence process in these materials and guide future experimental research.

4.1. Model description

Two slightly different models were used. The models were based upon work published elsewhere (Bøtter-Jensen et al., 1995; McKeever et al., 1997a,b; McKeever and Chen, 1997; Chen and McKeever, 1997; Bailey, 2001). All of these models simulate luminescence from a crystal by considering a valence band, a conduction band, trapping states within the band gap, and recombination centers within the band gap. With this type of numerical modeling, there are two basic approaches: (i) attempts to model overall properties and characteristics and to understand certain phenomena using a generalized model not meant to correspond to a specific mineral (Bøtter-Jensen et al., 1995; McKeever et al., 1997a,b); or (ii) attempts to develop a particular model that corresponds to the trap and recombination structure of a specific mineral (Bailey, 2001). The current work generally follows the first approach and does not attempt to model all the characteristics of the Mars simulant materials studied experimentally. Thus, the purpose of the model is to establish principles only and is not to provide a detailed description of the OSL characteristics of the materials under study. We intend to use the model only to teach us about possible behavior and to generate a general understanding of potential OSL characteristics.

The models used for this study consist of 4 or 5 traps and one recombination center. Real minerals have more than one recombination center, but the purpose of this study is to determine the effects of low-temperature traps without the added complexity of multiple recombination centers. As illustrated in Fig. 7, the electron traps consist of: (a) a low temperature, optically active trap with

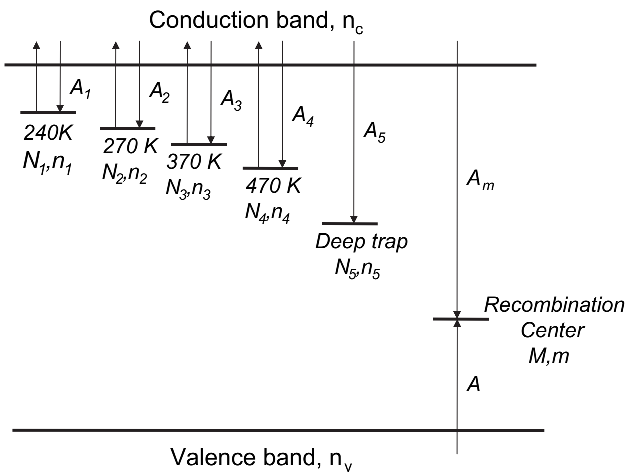

Fig. 7. Band diagram representing the traps and recombination center for numerical modeling of low-temperature traps and the OSL process. See Table 3 for the parameters used.

a TL peak near 240 K; (b) a trap with a TL peak around 370 K that is not optically active to simulate moderately shallow traps that act as competitors for charge but that are not directly involved in the OSL process; (c) the "main dosimetric trap" that is optically active (i.e. gives rise to OSL) and has a TL peak near 470 K; (d) a deep, thermally disconnected trap; and (e) an optically active trap with a TL peak near 270 K (for Model A only). The hole trap, acts as the recombination center. The numerical values chosen for the trapping and recombination parameters have largely been based upon work by Bøtter-Jensen et al. (1995) and McKeever et al. (1997a), but the energy depth (E) and frequency factors (s^{-1}) have been adjusted so that the TL peaks are in positions that broadly match those observed experimentally for each simulant. The parameters for the two models are given in Table 3. The energy levels in Fig. 7 and Table 3 are labeled according to the approximate position of the corresponding TL peaks (i.e. *240 K, 270 K, 370 K* and *470 K*) or according to their function (*Deep*, *Recombination Center*). As noted, the main difference between the two models is the presence of an additional optically active trap near 270 K in Model A. In terms of TL peak positions, the simulated TL curves roughly correspond to those from the two mineral mixtures used in this study, with Model A corresponding to OSU Mars-1 and Model B corresponding to OSU Mars-2.

The numerical modeling operates by solving a set of simultaneous, non-linear differential rate equations that describe the flow of charge in the system during all aspects of the luminescence process (i.e. irradiation, relaxation, heating, and optical stimulation). The equations assume that electron transport is via the conduction band, and that the crystal is a closed system (i.e. electrons are not lost to other processes). The rate equations used for modeling the luminescence process with i traps and one recombination center are:

$$\frac{dn_c}{dt} = f + \sum_i n_i f_2 + \sum_i n_i s_i \exp\left(\frac{-E_i}{k_B T}\right) - n_c \sum_i (N_i - n_i) A_i - n_c m A_m \tag{2}$$

$$\frac{dn_i}{dt} = -n_i f_2 - n_i s_i \exp\left(\frac{E_i}{k_B T}\right) + n_c (N_i - n_i) A_i \tag{3}$$

$$\frac{dm}{dt} = n_v (M - m) A - n_c m A_m \tag{4}$$

$$\frac{dn_v}{dt} = f - n_v (M - m) A \tag{5}$$

$$T = T_0 + \beta t \tag{6}$$

In these equations f is the radiation ionization rate (i.e. the electron–hole pair production rate, $cm^{-3}\,s^{-1}$, proportional to dose rate), n_i is the concentration of electrons in the ith trap (cm^{-3}), f_2 is the optical excitation rate (s^{-1}, same for both optically active traps, proportional to the stimulation light intensity multiplied by the photoionization cross-section), s_i is the frequency factor for the ith trap (s^{-1}), E_i is the trap depth of the ith trap (eV), k_B is Boltzmann's constant, T is the temperature (K), n_c is the concentration of free electrons (cm^{-3}), n_v is the concentration of free holes (cm^{-3}), N_i is the concentration of defects (available traps) for the ith trap (cm^{-3}), m is the concentration of trapped holes (cm^{-3}), M is the concentration of available hole traps at the recombination center (cm^{-3}), A_i is the electron trapping probability of the ith trap (cm^3/s), A is the hole trapping probability of the recombination center (cm^3/s), A_m is the electron–hole recombination probability at the recombination center (cm^3/s), t is time (s), and β is the heating rate (K/s). Also note that the recombination center is thermally stable. The above equations can be further simplified by making the usual quasiequilibrium assumption (Chen and McKeever, 1997; Bøtter-Jensen et al., 2003). Mathematically, this assumption is:

$$\left|\frac{dn_c}{dt}\right| \ll \left|\frac{dn}{dt}\right|, \left|\frac{dm}{dt}\right| \quad \text{and} \quad n_c \ll n, m \tag{7}$$

Physically, these inequalities mean that the electron population of the conduction band changes very little compared with the changes in the trap population. We can therefore assume that electrons never accumulate in the conduction band during the luminescence process and:

$$\frac{dn_c}{dt} \cong 0 \tag{8}$$

Since there is only one recombination center, the luminescence intensity (TL, or OSL depending upon the stimulation method) signal is given by:

$$I = -n_c m A_m \tag{9}$$

It is important to note that this expression for the luminescence intensity does not contain any explicit dependence on temperature. In other words, thermal quenching of the luminescence center is not considered at this time in the model. The first goal is to focus on the influence of the low-temperature traps.

Table 3
Parameters for the 5 traps and 1 recombination center used in the numerical simulations of low-temperature traps and the OSL process. See text for the equations used.

Level	N (or M) (cm^{-3})	E (eV)	s (s^{-1})	A (cm^3/s)	A_m (cm^3/s)
240 K[c]	5×10^{10} [a,b]	0.645 [a,b]	5×10^{12} [a,b]	5×10^{-10} [a,b]	–
270 K	1.5×10^{9} [a]	0.74 [a]	5×10^{12} [a]	5×10^{-10} [a]	–
370 K	3.5×10^{10} [a] 1.19×10^{9} [b]	1.0175 [a,b]	5×10^{12} [a,b]	5×10^{-10} [a,b]	
470 K[c]	2.5×10^{11} [a,b]	1.41 [a,b]	10^{14} [a,b]	10^{-10} [a,b]	–
Deep	5×10^{11} [a,b]	–		10^{-10} [a,b]	–
Lumin. Center	5×10^{12} [a] 5×10^{13} [b]	–		4×10^{-10} [a,b]	2×10^{-9} [a,b]

Other parameters: $f = 1 \times 10^8\ cm^{-3}/s$ (irradiation (ionization) rate); $f_2 = 1 \times 10^{-2}\ s^{-1}$ (optical excitation (ionization) rate); $\beta = \pm 5$ K/s (heating rate).
[a] Denotes the parameter values for Model A.
[b] Denotes the parameter values for Model B.
[c] Optically active.

The rate equations can be numerically solved for any of the operations involved in the luminescence process by changing the values of certain parameters: (a) for irradiation, $f > 0$, $f_2 = 0$, $\beta = 0$; (b) for relaxation (after irradiation or stimulation), $f = 0$, $f_2 = 0$, $\beta = 0$; (c) for heating, cooling, or TL, $f = 0$, $f_2 = 0$, $\beta = \pm 5$; (d) for bleaching or OSL, $f = 0$, $f_2 > 0$, $\beta = 0$.

The numerical modeling was carried out by solving the rate equations using *Mathematica* Version 4.2.1.1 (Wolfram Research, Inc.). The software was used to determine the basic luminescence characteristics of the model, simulate the SAR procedure (including dose recovery and equivalent dose (D_e) estimation) at normal terrestrial temperatures, and simulate the SAR procedure at martian temperatures. The irradiation and stimulation temperatures can be varied during the dose recovery or D_e estimation process for both models.

4.2. The model

Initially, all populations (n_v, n_c, $n_1 \ldots n_5$, and m) were set to zero to correspond to crystallization or formation of the crystal. Ionization (cf. the "dose") of 10^9 cm^{-3} electron–holes pairs was then simulated at a rate of 6.3×10^{-3} cm^{-3}/s to simulate the natural ionization rate, corresponding to 10^5 years of natural irradiation. Bleaching for 10^4 s was then included to simulate a period of natural sunlight bleaching. This in turn was followed by three cycles of irradiation at natural dose rates and periods (6000 s) of bleaching, to represent several bleach and burial cycles. This simulation of the natural "history" of the specimens was performed before every calculation presented in this work.

The TL curve predicted by the models was then examined. In fact, TL was used in development of the models in order that parameters (mainly E_i and s_i for the various traps) could be adjusted to produce TL peaks in approximately the desired positions. Since the position of the low-temperature peaks is important, the previously described geologic history was carried out at 173 K. TL then was calculated while heating from 173 K to 773 K at a heating rate of 5 K/s using a "laboratory" ionization rate of 10^8 cm^{-3}/s. The results of this calculation are shown in Fig. 8, which shows the entire TL curve ((a) and (b)) and the range from 173 K to 273 K in detail ((c) and (d)) for both models. The TL curves show peaks at approximately 240 K, 270 K (for Model A), 370 K and 470 K, and these peak positions led to the labels used in Fig. 7 and Table 3.

The sensitivity changes produced by the model from repeated cycles of irradiation, pre-heating, and OSL measurement were then examined. After simulating the geologic history at 298 K, the degree of sensitivity change was then found by performing 10 cycles consisting of: (1) net ionization of 2×10^7 cm^{-3} by irradiation at 298 K; (2) a preheat at 373 K for 10 s; and (3) OSL for 600 s at 398 K. The resulting OSL signals increased by 38% for both models over the 10 cycles for the 1 s signal (sum of the intensity from the first second minus the 1 s average of the last 5 s of stimulation). This amount of sensitivity change is certainly less than that displayed by either feldspars (Blair et al., 2005) or quartz (Murray and Wintle, 2000). However, the mechanisms generally believed to be the cause of sensitivity change, strong competition during irradiation and stimulation for feldspars (Duller, 1997) and mobility of holes during heating of quartz (Murray and Wintle, 2000), are not included in this model and thus the lack of displayed sensitivity change is not surprising.

The next step in characterization of the models was to construct OSL dose response curves. Again, after simulating the geologic history (at 298 K), a preheat at 373 K for 10 s and a bleach for 600 s at 398 K were administered. Dose response curves were then constructed using a regenerative-dose procedure without

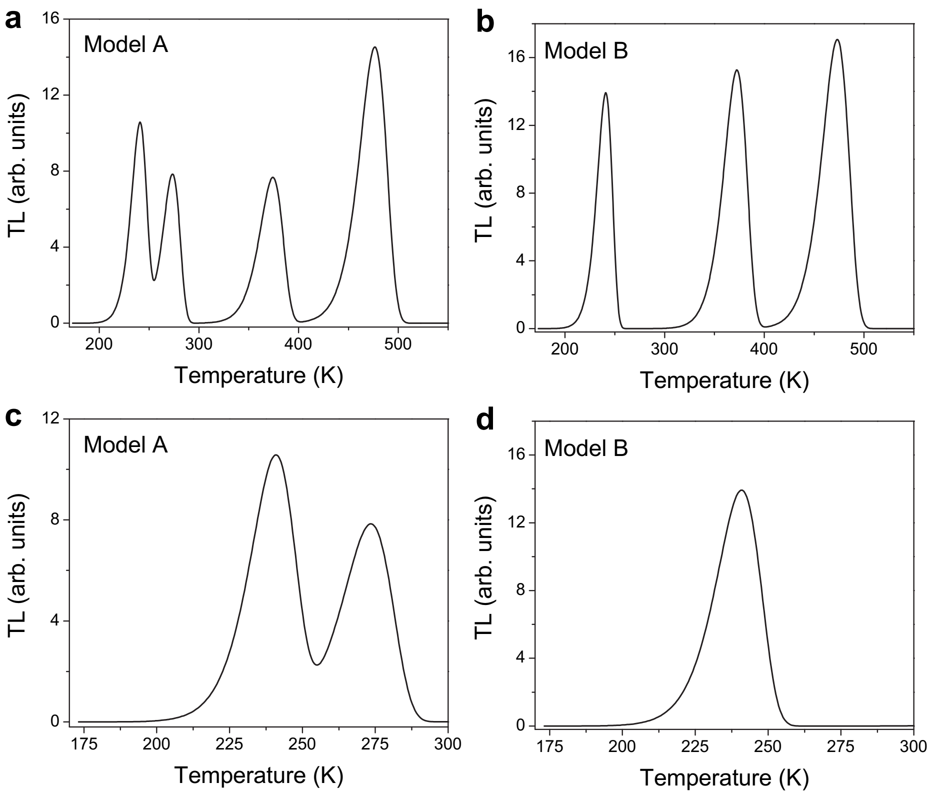

Fig. 8. TL from Model A ((a) and (c)) and Model B ((b) and (d)). The geologic history was simulated at a fixed temperature of 173 K, and a dose delivered at a laboratory dose rate was then given. The TL was calculated while heating from 173 K to 273 K at a heating rate of 5 K/s. Graphs (a) and (b) show the entire TL curve, while (c) and (d) show the curve only from 173 K to 298 K.

a sensitivity correction. Irradiations were at 298 K, followed by a pre-heating at 373 K for 10 s, and OSL measurements for 600 s at 398 K. The results showed a linear dose response curve for both models, up to the onset of saturation as the total ionization approached the trap concentrations used in the model (Table 3). Slight supralinearity due to sensitization was observed for both models before the start of sublinear growth.

Several simulations were run to compare the behavior of the models and soil simulants when irradiations and/or OSL measurements are performed at low temperatures. The irradiation temperature was kept at 298 K while the stimulation temperature was varied from 298 K to 173 K, corresponding to the data of Fig. 4. The results are shown in Fig. 9. Both models show an initial increase in OSL as the stimulation temperature is lowered due to the *370 K* traps beginning to contribute more strongly to the OSL signal. The OSL signal then decreases as the *240 K* traps capture charge during stimulation. This effectively makes the recombination process less efficient. Overall, however, the models do not show a strong dependence on stimulation temperature, unlike the experimental results, because thermal quenching is not included.

The experiment in which the irradiation was performed at various temperatures between 298 K and 173 K and the stimulation temperature was fixed at 298 K (Fig. 5) was also simulated. The results from the simulations are shown in Fig. 10. OSU Mars-1 shows a strong decrease followed by a slight increase as the irradiation temperature is lowered. However, OSU Mars-2, Model A and Model B all show an increase in OSL with decreasing temperature; indeed both models closely approximate the characteristics of OSU Mars-2. In the models, the *370 K* and *240 K* traps begin trapping charge as the temperature is lowered, and a portion of this charge is then transferred to the *470 K* trap during heating to 298 K. The OSL signal then increases. However, why OSU Mars-1 does not show this same behavior is not clear.

As a final simulation, the experiment leading to the data of Fig. 6 was simulated using various irradiation and stimulation temperatures from 298 K to 173 K. The results from both models are shown in Fig. 11. The model results are qualitatively similar to the results from the Mars mixtures although the models show more dramatic increases and decreases.

4.3. Dose recovery and dose estimation at terrestrial temperatures

A dose corresponding to a total ionization of 10^9 cm^{-3}, at either laboratory or natural dose rates, was given after first simulating the

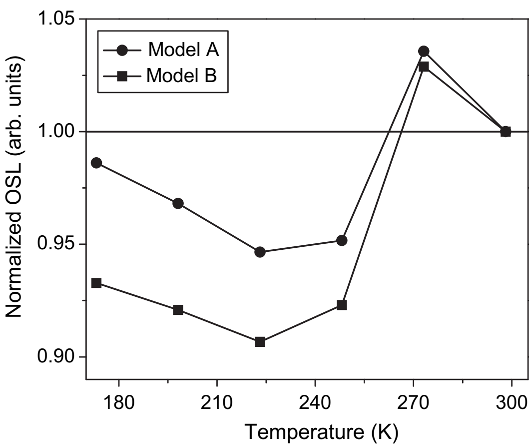

Fig. 9. OSL from the models with irradiation at 298 K and OSL stimulation at various temperatures as described in the text.

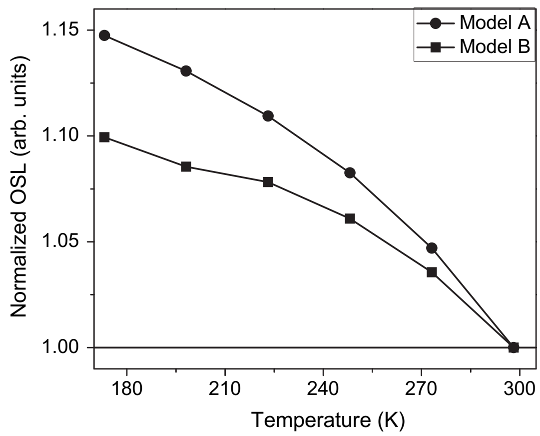

Fig. 10. OSL from the models with irradiation at various temperatures and OSL stimulation at 298 K as described in the text.

geological history of the sample (see previous section). Then, the SAR procedure was used to either recover the laboratory dose or estimate the natural dose. The parameters of the SAR procedure were: a preheat at 473 K for 10 s after both the regeneration dose and the test dose; OSL measurement at 398 K for 600 s; a small test dose at 298 K; and regeneration doses of 8×10^8, 10^9, 1.2×10^9, and 8×10^8 (repeat point) cm^{-3} total ionization delivered at 298 K. The test dose was the equivalent of a total ionization of 2.5×10^8 cm^{-3}. The dose recovery ratio after delivery of a known laboratory dose was 1.00 for both Models A and B, indicating that a known dose can be satisfactorily recovered under these conditions.

4.4. Dose recovery and dose estimation at martian temperatures

A series of simulations was undertaken to investigate what laboratory irradiation temperatures and OSL stimulation temperatures are optimal for recovering or estimating a dose delivered at 173 K. For these simulations, the geologic history was simulated at 173 K. As before, the same natural doses and laboratory doses were applied and the SAR procedure was simulated to recover these doses. However, in these simulations, the parameters of the SAR procedure were varied in order to determine how low-temperature traps affect the dose recovery process. The tested parameters of the

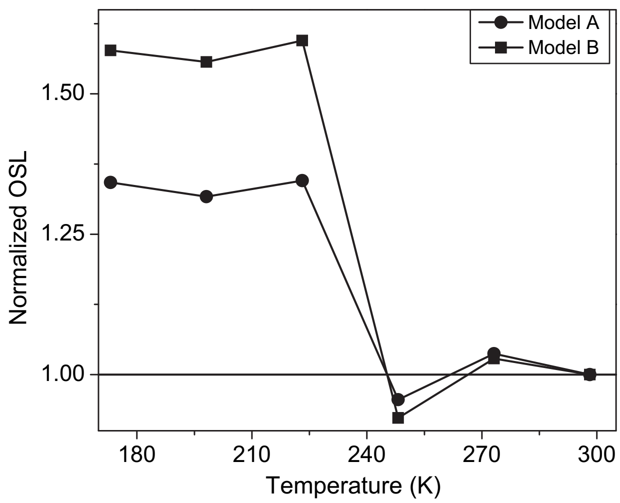

Fig. 11. OSL from the models where the irradiation and stimulation temperatures have been varied together.

Table 4
Results of dose recovery and dose estimation simulations with both models. In these simulations, the laboratory or natural dose was delivered at 173 K and irradiation and stimulation were carried out at the specified temperature. "Lab" refers to a dose (i.e. the dose to be determined) delivered at high laboratory dose rates, whereas "Natural" denotes a dose delivered at low natural dose rates.

Dose	Irr. *T* (K)	OSL *T* (K)	Dose ratio	
			Model A	Model B
Lab	173	173	0.99	0.99
Lab	173	298	1.00	1.00
Lab	298	298	1.00	0.99
Natural	173	173	0.75	0.60
Natural	173	298	1.00	1.00
Natural	298	298	1.00	1.00

SAR procedure were: (a) laboratory irradiation and stimulation at 173 K; (b) laboratory irradiation at 173 K and stimulation at 298 K; and (c) laboratory irradiation and stimulation at 298 K. It is important to note that no preheats were used in these simulations (for the rationale discussed earlier).

The results of the dose recovery experiment are given in Table 4 for both Model A and Model B. For both models the known dose can be recovered with negligible errors by any of the three methods. These results are consistent with the measured values for OSU Mars-1 and Mars-2 given in Table 2. While these results indicate significant flexibility in the choice of measurement parameters, the models were further tested by attempting to recover natural doses delivered at 173 K.

Simulations with the same natural dose were carried out for both models. The results of these simulations are again given in Table 4. For both models there is a significant underestimation of the natural dose if both the calibration irradiations and OSL measurements are performed at 173 K (dose ratios of 0.75 for Model A and 0.60 for Model B). This is to be expected since the *240 K* traps are not effectively populated during natural irradiation (the lifetime of the trap is small compared to the irradiation time) and therefore do not contribute to the natural OSL signal. However, these traps are populated during laboratory irradiations at 173 K, which results in proportionally larger OSL signals and a subsequent underestimation of the natural dose. However, the natural dose can be accurately estimated by measuring OSL at 298 K and irradiating for calibration at either 173 K or 298 K.

5. Discussion and conclusions

The expansion of OSL dating into the martian environment, where sediments may have been stored and irradiated at low temperatures, is an exciting possibility. The influence of low-temperature traps (those with peak temperatures below room temperature) and the luminescence efficiency below room temperature are largely unknown for most materials. The experiments and simulations in this paper attempt to understand some of the effects a low ambient temperature could have on luminescence from martian soil simulants.

The luminescence process can be thermally dependent for several reasons, including low-temperature traps and the stability of these traps over geological time scales, thermal assistance in the stimulation process, and thermal dependence of the recombination process and luminescence efficiency. Some of these properties were studied for OSU Mars-1 and OSU Mars-2 using a low-temperature OSL system. It was found that low-temperature optically active traps are present in these materials, the luminescence efficiency is thermally dependent, and the OSL process is dependent upon both the irradiation and the OSL measurement temperatures. These results were used to develop some general models of the luminescence process when low-temperature traps are present.

Two models, named Model A and Model B, were developed so that the TL and OSL processes could be simulated. With the notable exception of the thermal dependence of the luminescence efficiency, the two models can qualitatively account for the observed behavior of the minerals. Although Model A contains an additional low-temperature TL trap, the two models produce similar results under the tested conditions. Both models were able to recover either a laboratory or a natural dose delivered at 173 K if the OSL measurement temperature was maintained higher than the maximum temperature during irradiation. Similar observations were reached by Kalchgruber et al. (2007) who performed similar model simulations using the same model but with different model parameters.

The absence of thermal quenching from the model simulations has been noted. However, thermal quenching is only a factor when simulating OSL at different stimulation temperatures. Simulations of the OSL variation as a function of irradiation temperature, for example, are unaffected by this omission. Thus we observe that the simulations of Fig. 10 show pleasing similarity with the results for OSU Mars-2 in Fig. 5(b). OSU Mars-1, however, shows a notably different behavior (Fig. 5(a)). The differences in the TL from the two simulant samples is only in the addition of a trap near *280 K* for OSU Mars-1 and it is unlikely that this alone is sufficient to explain the different OSL characteristics of this material. Mineralogically the differences are in the reduced amount of plagioclase and pyroxene and the addition of obsidian in Mars-2 compared with Mars-1. The significance of this will need to be investigated elsewhere.

Nevertheless, despite these differences in the two simulant samples it was found that known doses delivered at either 298 K or at 173 K, and also when doses were delivered stepwise at three different temperatures, could be accurately recovered as long as the OSL measurement temperature was equal to or greater than the maximum temperature experienced during delivery of the dose. The same conclusions were reached whether simulating the OSL using Model A or Model B. The same conclusions were also reached by Kalchgruber et al. (2007). Thus, although all OSL measurements must be performed at the same fixed temperature in order to avoid changes in the luminescence efficiency (e.g. Figs. 2 and 4) measuring OSL at the lowest temperature and highest efficiency is not advisable. As discussed previously, any unstable low-temperature traps will need to be emptied during a dating procedure. This situation is similar to the influence of the *380 K* trap in quartz OSL dating (Murray and Wintle, 2000) where OSL is measured at an elevated temperature (e.g. 398 K) to keep the *380 K* trap empty even though the signal is somewhat reduced by thermal quenching.

The simulations of Section 4 serve to emphasize the potential importance of low-temperature traps in the OSL process. It was shown (Figs. 9–11) that much of the behavior of the martian soil simulants can be qualitatively explained solely through the competition between optically active low-temperature traps and optically insensitive traps. Both models also showed that shallow traps can create significant competition effects during irradiation and stimulation procedures.

The presence of trapping states with optical sensitivity below room temperature (Fig. 3) is significant for dating applications. These states may not be stable over geologic time scales (even in colder environments) and therefore may not contribute to the natural signal, or do so only weakly. However, if laboratory irradiations were performed at low temperatures, these states would be populated and therefore would contribute to subsequent calibration OSL measurements. The simulation results for both models show this in that the OSL stimulation temperature cannot be at the same temperature as natural irradiation. Either the irradiations need to be performed at higher temperatures to keep the traps empty, or the samples need to be warmed before OSL measurement in order to minimize the influence of the low-temperature traps. The potential of shallow traps (not necessarily low-temperature traps) to affect the luminescence process in feldspars has long been recognized when using blue or green stimulation (Duller and Bøtter-Jensen, 1993), and it has been suggested that OSL measurements should be made at elevated temperature. For infrared stimulation, Poolton et al. (2002a,b) found that shallow traps had little effect on the OSL signal and concluded that elevated stimulation temperatures should not be used as the stimulation could empty traps not emptied under natural bleaching conditions. The effects of shallow traps and elevated stimulation temperatures have not been fully investigated for a post-IR blue stimulation procedure (Banerjee et al., 2001; Blair et al., 2005). However, the low-temperature system described in this work does not currently have the capabilities to test the post-IR blue stimulation sequence.

Taking into account the experimental results along with the results of the modeling work, it is recommended that OSL dating procedures for martian sediments be adopted (and an instrument be designed to carry out these procedures) that utilize OSL measurement at temperatures higher than the ambient temperatures experienced in nature to reduce the influence of any unstable low-temperature traps in the luminescence process. These experiments did not investigate the effects of pre-heating the samples and no specific pre-heating temperatures or procedures can be suggested based upon the current findings. However, OSL measurement at higher temperatures than irradiation reduces the importance, and perhaps the necessity, of specific pre-heating procedures since the results also show the potential for OSL dating of the martian sediments without the need for pre-heating.

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